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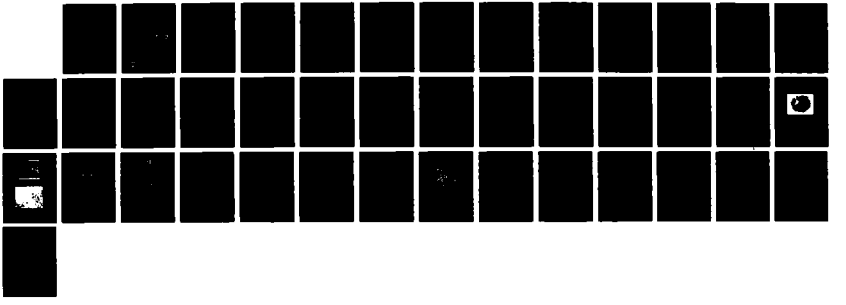

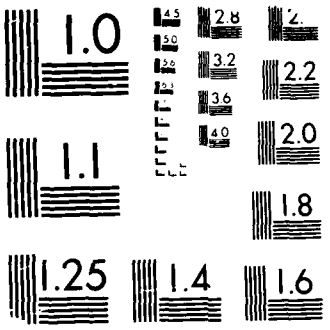

MICROCOPY RESOLUTION TEST CHART

UCLA School of Engineering and Applied Science

Effect of the Matrix on the Creep-Behavior of Carbon-Carbon Composite

by George H. Sines, Zheng Yang, Brian D. Vickers

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L.H.Peebles, Monitor

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UCLA SCHOOL OF ENGINEERING AND APPLIED SCIENCE

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Creep specimens and experiments were designed to determine the properties of carbon-carbon composites at high temperatures (over 2310°C) and at high stress levels (over 770 MPa). During the course of these experiments it was observed that the carbon matrix greatly increases the creep resistance of carbon fibers in properly impregnated, uniaxial composites. Results show how a composite specimen creeped 3.6% after 5.9 hrs. of testing while a matrix-free specimen, at the same test conditions, ruptured with 138% strain after only 0.39 hrs. . This improvement is attributed to the alignment of the mesophase layers of the carbon matrix, which distribute tensile loads more evenly amongst the filaments and which may also alter the filaments' stress state by restricting transverse strains. Also of interest was the large primary creep response associated with the composite at these high stress and temperature levels. Because it was such a large percentage of the entire response, this initial,

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ABSTRACT

Creep specimens and experiments were designed to determine the properties of carbon-carbon composites at high temperatures (over 2310 C) and at high stress levels (over 770 MPa). During the course of these experiments it was observed that the carbon matrix greatly increases the creep resistance of carbon fibers in properly impregnated, uniaxial composites. Results show how a composite specimen creeped 3.6% after 5.9 hrs. of testing while a matrix-free specimen, at the same test conditions, ruptured with 138% strain after only 0.39 hrs. This improvement is attributed to the alignment of the mesophase layers of the carbon matrix, which distribute tensile loads more evenly amongst the filaments and which may also alter the filaments' stress state by restricting transverse strains. Also of interest was the large primary creep response associated with the composite specimens at these high stress and temperature levels. Because it was such a large percentage of the entire response, this initial, transient creep should become the main design parameter of any attempt to relax bundle stresses in the fabrication of carbon-carbon billets.

ACKNOWLEDGEMENTS

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INTRODUCTION

Cylindrical weave, carbon-carbon composites have successfully been utilized as integral throat and entrance components for advanced rocket nozzle applications. Possessors of unique thermo-mechanical properties, their use has improved the reliability and performance of these systems. However, problems have occurred during their fabrication as a number of carbon-carbon composite billets have either fractured or developed severe structural anomalies [1]. Generally accepted as responsible for these problems are the high thermal stresses caused by repeated heatings to the high graphitization temperatures of processing [2,3].

The emergence of advanced composite materials over the past twenty years is well-documented, [eg. see Fitzer, ref. 4, for a thorough review of carbon-carbon composites]. In the area of carbon-carbon research a great deal of effort has been spent in trying to understand what happens to these composite billets during their fabrication. Investigators have looked at fiber and matrix properties [5,6], stress distributions in the billet during heating and cooling [2,7], failure mechanisms and crack morphology [1,3] etc ... Our group has been focusing on the creep of carbon-carbon composites at high stress levels and at high temperatures [7,8], as these are the conditions which the billet encounters during its fabrication. The quantitative information derived from our study is needed so that time-temperature processing paths might be designed which would minimize thermal stresses via creep. During the course of experiments developed to measure carbon-carbon composite creep, some interesting qualitative observations were made regarding the role of the carbon matrix on the creep-behavior of the carbon yarn.

Creep studies of carbon-carbon composites are few, [refs.7,8,9,10,11,12,13 are all that we are aware of], and they do not consider how the two carbon phases interact. As

for advanced composites in general, a fairly extensive amount of theoretical and experimental work on creep-behavior is available, [see refs. 14 and 15 for example], but again, attention given to the interactions of the composite's constituents is lacking. Typically, the creep behavior of advanced composites is modeled in the following ways: For composites with discontinuous fibers the matrix usually assumes the role of limiting factor because its melting point is generally lower than the fibers. For continuous composites, two cases are considered: that of rigid fibers in a creeping matrix and that of creeping fibers in a creeping matrix [14]. These models, however, do not seem applicable to carbon-carbon composites.

With the above serving as a backdrop, this paper reports that the carbon matrix substantially improves the creep resistance of the carbon fibers and that this improvement is a strong function of the degree of interfacial bonding. In conjunction with reporting these findings, the testing methods and specimens developed to measure creep in uniaxial, carbon-carbon composites are also described. These methods and specimens are being used in a study in progress on the activation energy and stress exponent of creep.

EXPERIMENTAL

The proper design and preparation of the experiment's creep specimens was vital if meaningful data were to be gathered. The design of the specimens had to satisfy these two requirements: that the specimens be strong enough to permit testing at high stress levels and that an accurate gage length be established. There was also two important factors regarding the specimens' preparation: that there be adequate impregnation and interfacial bonding of the carbon yarn by the carbon matrix, and that oxidation be avoided during the pyrolysis process. These procedures, which are about to be described, are derived from the work done by Quan [7] and Mack [8], with several modifications made by the authors.

Graphite fiber yarn, as received from Hercules Incorporated, (designated as HMPVA-3K with 3000 PAN-based graphite filaments per yarn; nominal filament diameter of 7 microns), were looped and cut to size, (as shown in fig.1), with a single yarn extending the length of the specimen. Cotton ties were used to hold the specimen's shape during pyrolysis as their organic composition allowed them to maintain their strength at high temperatures in an inert atmosphere before turning to carbon. The six-yarn reinforced sections, which defined the one inch test section, formed strong loops at the ends which permitted testing at high stress levels. The specimens were further reinforced by two small graphite blocks which prevented the end loops of the impregnated graphite yarn from splitting open while under load.

Processing these tied, graphite fiber specimens into carbon-carbon composite test specimens involved the following series of steps. First, the as-received yarn had a protective sizing on it which was found to greatly hinder matrix bonding if not properly removed. The most effective method was one which thermally removed the sizing from the

yarn at a temperature of 350 C for ten minutes in an inert atmosphere.

Coal tar pitch, (produced by the Allied Chemical Corp. and designated as CP277-15V), was the medium used as matrix precursor. This pitch was first preheated at a temperature of 300 C for approximately two hours in a nitrogen atmosphere to drive off many of the volatiles and impurities contained within. It was then finely ground and used as impregnate.

Impregnation of the specimen was done at a temperature of 250 C in a nitrogen atmosphere in a horizontal tube furnace. The tied specimens were placed in a steel trough, buried beneath a mound of ground pitch, and then were slowly wetted by the impregnate which melted with the rising temperature. After approximately three hours of immersion in this molten pitch bath, the specimens were removed, gently stretched so as to straighten and then carbonized.

Carbonization of the impregnated specimens was done by heating to a temperature of 900 C in a nitrogen atmosphere. (The carbonization schedule is shown in fig. 2.) Heating occurred over a 58 hour period, during which the coal tar pitch slowly passed through its mesophase state (about 450 C) as it hardened. A slow heating rate was needed to limit the percolation of any residual pitch volatiles within the specimen so that distortions of the matrix microstructure from bubble pores might be restrained. Completion of the carbonization process yielded uniaxial, carbon matrix-carbon (graphite) fiber, creep specimens ready to be graphitized and tested.

The preparation of the creep specimens in this manner yielded sufficiently dense specimens with well-bonded filaments. (This judgment was supported by simple four point bend tests performed on trial specimens - they fractured brittlely.) It should be noted that only one impregnation-carbonization cycle was necessary to achieve proper densification. This is in contrast with the fabrication of a typical carbon-carbon billet which goes through several high pressure cycles to fill cooling cracks and to replace volatilized material [3]. The relatively

small scale of the test specimens along with the absence of transverse fibers and matrix pockets helps to account for this processing advantage. Cross sections of our test specimens are shown in fig. 3.

That only one combination of fiber and coal-tar pitch was used for the test specimens should not effect the generality of the results as far as the influence of the matrix on the yarns' creep-behavior is concerned. This assertion is supported by White and Sheaffer [16] who observed consistent patterns of wetting and mesophase alignment regardless of fiber type. Their work also showed that the pressure during the pyrolysis process had little effect on the matrix's microstructure, with bloating still active in the matrix even at 15 ksi. (The pressure on our test specimens were slightly above atmospheric and some bubble pores were discovered.) Finally, rupture tests performed by Mack [8] on similarly prepared specimens had tensile strength values between 185 and 260 ksi at room temperature.

To show how the matrix affects the fiber's creep behavior, a specimen was needed with a test section composed of graphite yarn without the carbon matrix. This condition was partially achieved accidentally when a solvent, (methyl-ethyl-ketone, or MEK), was used to remove the sizing in place of the thermal method. But the MEK, instead of removing the sizing, only dissolve it and then leave behind a sizing residue on the specimens' surfaces as it evaporated. This residue would greatly inhibit impregnation with the resulting specimens left relatively free of any properly bonded matrix. This condition was further exacerbated when the MEK was reused several times; the result being a progressive increase of residue left in and on the specimens and increasingly poorer impregnation. The poor performance of the specimens prepared in this manner was the first indication of the strengthening effects that the carbon matrix could have on the creep resistance of properly impregnated graphite yarn.

To fully demonstrate this strengthening effect a specimen with a gage section completely free of matrix was required. To create such a specimen, special care had to be

taken during pyrolysis to prevent any fiber wetting during impregnation and any carbon vapor deposition during carbonization. This was accomplished by tightly covering the single yarn test section with a graphite casing, (see fig.4). By using this casing the rest of the specimen - namely its reinforced sections - could still be properly processed so that it would be strong enough to sustain the test loads.

The testing apparatus used for these experiments centered around an electric resistance, graphite element furnace, (from Astro Furnace, model number 2570), which we modified for our purposes. The graphitization of the specimens and their subsequent creep testing were performed in an inert atmosphere, helium, to avoid any oxidation of the heating elements, fixtures and specimens. (Testing in an Argon atmosphere suffered from arcing at high temperatures.) An optical pyrometer, (A Honeywell Radiamatic, type R13), was used to control and measure temperature. A linear variable differential transducer, (or LVDT, from Schaevitz Engineering, model number 200DC-D), measured changes in length, whether they be the result of temperature, of applied loads, or of creep. A diagram of the furnace and its features are shown in fig. 5.

Test loads were applied to a specially designed loading beam which pivoted on two knife edges, (see fig. 6 for details). This loading beam, anchored to the base of the furnace shell by a hanging frame, was supported in a manner which prevented any lateral or twisting motions. Its design had several advantages: the test specimens could be loaded without shock via the slow release of the restraining screw which supported the beam; the specimens were also prevented from swinging in the furnace during testing and during load changes so the optical pyrometer's focus could not be disturbed; and the LVDT, because it could be mounted on the beam - and away from the loading axis, made the equipment more accessible, easier to adjust, and more sensitive since beam displacement at the LVDT's location was a magnification of the specimen's elongation.

The declaration that the creep specimens had a knowable gage length is not a trivial pronouncement. To legitimize

this one inch, gage length claim, two important test conditions had to be accounted for: the temperature and the stress distribution. First, the temperature profile of the furnace was approximated as parabolic. Its hot zone, centered on each specimens, test section, was assumed to remain relatively constant in temperature over the length of the test section, (a reasonable approximation according to the temperature distribution analysis done by Feldman, ref.11). Regarding stress, since the cross-sectional area of the specimens' reinforced sections were six times greater than that of their test sections, the stress experienced by the test sections was necessarily six times greater. When one includes a knowledge of the stress exponent, (this value, as determined from our creep results so far, appears to fall in the range of 7 to 8), the assumption that creep was confined to the one inch test section was quite reasonable. Verification came from creep tests performed on specimens without a test section, - i.e., the specimens had a cross section of six yarns along their entire length. These specimens showed no elongation when tested at load and temperature levels which did generate creep in specimens with one bundle test sections.

The creep experiments were performed in the following manner: First, each test specimen was vertically supported in the furnace by two pin and clevis test fixtures which were machined from graphite blocks. This assembly was contained within the furnace, sealed, and then the atmosphere inside the furnace was purged with helium. After the initial purge, a steady helium pressure of approximately two inches of water was maintained for the duration of the test. An initial pre-load of 227 grams was hung from the loading beam and supported by the specimen while the furnace was manually controlled to the desired test temperature. Thermal expansion of the specimen and of the furnace and fixtures was measured by the LVDT during the heating period. When the test temperature was reached, deemed stabilized, and held for approximately 20 mins., the test load was applied - first to the loading beam and then slowly to the specimen by lowering the restraining screw. At this time, (t=0 in the graphs), creep began to be recorded as elongation vs. time. Graphitization of the specimen, dependent on both stress and temperature, occurred during

the heat up and the holding; there may have also been a small amount of graphitization after the application of the test load. (Temperature fluctuations during testing were estimated as 1 to 5 C and were quickly corrected manually when they occurred.)

RESULTS AND DISCUSSION

Figure 7 details the results from tests conducted on three different types of specimens: a fully impregnated, mono-yarn specimen as represented by curve 2C; a partially impregnated (MEK treated) specimen as represented by curve 5b; and two 'dry' specimens, curves F3 and F2, which represent specimens whose test sections consisted of a single, matrix free, graphite yarn. These four tests were all performed at the same test temperature of 2310 C (4190 F), and at the same test load of 9072 grams (20 lbs) which is equivalent to a net fiber stress of 770 MPa (112 ksi). The thermal response portion of the curve reflects the simultaneous effects of thermal expansion and graphitization of the specimens, and the thermal expansion of the furnace and fixtures. Likewise, the elastic response portion is the response of the specimens and fixtures to the applied load at t=0 hrs. Both of these responses of the four specimens were comparable, hence the representation by single curves.

What are not comparable are the specimens' creep responses. The matrix-free filaments of the 'dry' specimens' test sections stretched and ruptured quickly, showing no steady state behavior at these stress levels. Faring a little better was the partially impregnated specimen, as creep stages were apparent, albeit short-lived, with the strain at rupture approximately an order of magnitude less than its 'dry' counterpart. These results are in marked contrast with the composite specimen 2C. Whereas the 'dry' specimen had already ruptured after elongating approximately 138%, specimen 2C had at the same elapsed time strained only about 1% and was still in its primary creep stage . After 5.9 hours, 2C had elongated by only 3.6% and showed no signs of an impending failure. See table 1 for a numerical summary of the results. It should be noted that the reported 138% rupture strain for specimen F2 is an approximation, (despite the presentation of three significant figures), determined from ruler measurements after the ruptured sections were put 'back together'.

Still,a fraction of the yarn's filaments actually stretched about this amount before finally rupturing.

The above results may run counter to one's general preconception that continuous composites should be less creep resistant than their matrix-free fibers, (eg., see the illustrations of Lilholt, ref.14); especially when one takes into account the fact that the carbon yarn is the principal load bearing phase, (see ref. 4, pg. 164), and that the carbon matrix is quite brittle with many transverse cracks present. Unfortunately, (or fortunately depending on one's perspective), carbon-carbon composites do not readily lend themselves to these type of advance composite generalizations. This is due to the unique, formation properties of the carbon matrix - properties which help to distinguish it from other matrix materials.

This characteristic strengthening of the carbon fibers is attributed to the matrix microstructure of aligned carbonaceous mesophase - ie. the parallel orientation of the 'liquid crystal', graphite basal planes with the filament surfaces during pyrolysis [1]. This alignment creates a 'sheath effect' and, as reported by White and Sheaffer [6,16], this 'sheath effect' is the dominant formation mechanism of the matrix microstructure - being responsible for the interfacial bonding. The proper wetting and alignment of the mesophase during impregnation and carbonization improves the matrix's ability to graphitze and thus has a direct bearing on its ability to distribute forces between fibers (filaments) and, consequently, on the improved use of the composite [17].

Based on the above observations and the results of these creep tests,these microstructural arguments can be extended to the creep-behavior of carbon-carbon composites. The carbon yarn, when left 'dry' as specimens F3 and F2 were, was unable to properly distribute the testing loads thereby leaving individual filaments to creep and rupture haphazardly with the subsequent rapid creep failure of the entire yarn. This could be observed during the actual monitoring of the test as small, but perceptible, discontinuous jumps in elongation occurred in the recording. (This has also been observed by Feldman [11]. It should be

noted that impregnating the carbon yarn does not mean that the applied loads are perfectly distributed between the filaments, as these discontinuous jumps were also observed for the composite specimen - but they were less frequent and smaller.) Thus, stress transfer between the composite's constituents is regarded as an important element of carbon-carbon composite creep resistance.

Examinations of post-creep specimens offer some visual evidence supporting this conclusion. Scanning electron micrographs by Feldman [9], clearly show extensive filament necking in regions exposed by transverse matrix cracks. Our own photographs of post-creep, composite cross-sections, (see fig. 8), show how well-bonded filaments generally experience little or no diametral changes, (though the strains are quite small for these composite specimens anyway). It thus appears that unbonded filaments, exposed via cracks or by stress induced debonding, are much more susceptible to creep; that the matrix of well impregnated and bonded yarn has a cumulative strengthening effect across the specimen's thickness because of better load distribution between the filaments; and that yarn which is only partially impregnated, (curve 5b), is only partially strengthened.

Yet stress transfer may not be the complete story. In addition to the above discussion we propose that this 'carbon sheath' microstructure can also improve the carbon yarn's creep resistance by altering each bonded filament's stress state. The reasoning behind this assertion is fairly straightforward. The carbonaceous mesophase, when firmly layered on the filaments' surfaces, i.e., well-bonded, acts to constrain the transverse strains, thereby imposing a triaxial stress state. With a triaxial stress state filament plastic flow in the axial direction would be delayed until higher stress levels had been reached or debonding had occurred.

A simple analogy can be interjected here by considering a piece of candy - say some taffy - as representative of a carbon yarn filament. If one took a long narrow piece of taffy, heated it, and then pulled on it, it would stretch, neck and rupture without a great deal of effort. Now with another piece of this taffy, give its surface a thin hard

candy coating. Assuming that the properties of this coating do not change at the 'test' temperature, one is now required to pull much harder on this composite candy in order to stretch it since the taffy is now constrained from constricting by its coating. If this coating should suddenly crack during the 'test', a uniaxial stress state would be reintroduced, with necking and rupture again occurring quite readily. With this in mind consider the predicament of the 'dry' specimens. Their filaments, already unevenly loaded because of the absence of matrix, are further burdened by a uniaxial stress state because of the missing 'sheath effect'. It is thus not very surprising that these matrix-free specimens creep and rupture much more quickly than their properly impregnated counterparts.

Finally, there is some preliminary creep data to report as measured from curve 2C The steady state creep rate is measured as 0.00368 hr^{-1} with a total strain of 3.6% after 5.9 hours of testing. Of special interest from a processing perspective is the data gathered from the primary creep (stage I) portion of curve 2C. Approximately 2.75 hours had elapsed before steady state creep could be considered as the dominant feature. At this time the specimen had already elongated by 2.3% - a large percentage of its total measured elongation. (Indeed, after only one hour the specimen had creeped approximately 1.5%.) Other carbon-carbon composite creep investigators do not report observing such a large primary response. The explanation for this could be that the specimens they used could only be tested at low stress levels [7,9,11].

This considerable initial response should be considered if creep is to be used to relax stresses in the yarns of cylindrical weave billets during fabrication. (Maintaining the structural integrity of, say, the radial yarns is desirable because their presence improves the billets survival chances during its fabrication - although a recent analysis by Jortner [18] shows how damaged radials can still act to reduce the risk of billet failure.) In Quan et al. [7], analysis was based on a linear (steady state) creep

responses. In light of the above observations, this analysis should be reworked so that it takes advantage of the large primary creep stage.

CONCLUSION

In an effort to improve the understanding of the thermomechanical behavior of carbon-carbon composites the following was accomplished:

Testing apparatus and specimens were designed which permitted uniaxial creep testing at high temperatures (2310 C) and stress levels (112 ksi) - levels which are comparable to those experienced by carbon-carbon billets during their fabrication.

As a result of processing techniques developed by the authors, substantially dense carbon-carbon specimens with good bonding between matrix and filaments could be obtained in a single impregnation-carbonization-graphitization cycle without the use of an autoclave. This is in contrast to typical billet fabrication techniques which use several high pressure, high temperature, cycles.

During the course of on-going experiments to determine the stress exponent and creep activation energy the following observation was made: that the carbon matrix significantly improves the creep resistance of the carbon fibers. This contradicts the prevalent thought that the creep of fibers should be only slightly influenced by the matrix when the matrix's mechanical properties are inferior to those of the fibers.

This improvement in carbon fiber creep resistance can be attributed to the alignment of the carbonaceous mesophase layers in the matrix. This type of microstructure creates a 'sheath effect' which distributes loads more evenly and which may also impose a triaxial stress state in the filaments, and thus inhibit plastic flow.

The creep response of a composite specimen is noticeable for its rather large primary stage, as compared to its steady state response. The implications involved

here lie with the fabrication of carbon-carbon billets and the proposal that creep be used to relieve the stresses in the radial fibers. Instead of designing time-temperature processing paths on steady state creep predictions, more of the attention should be given to the initial, primary, response.

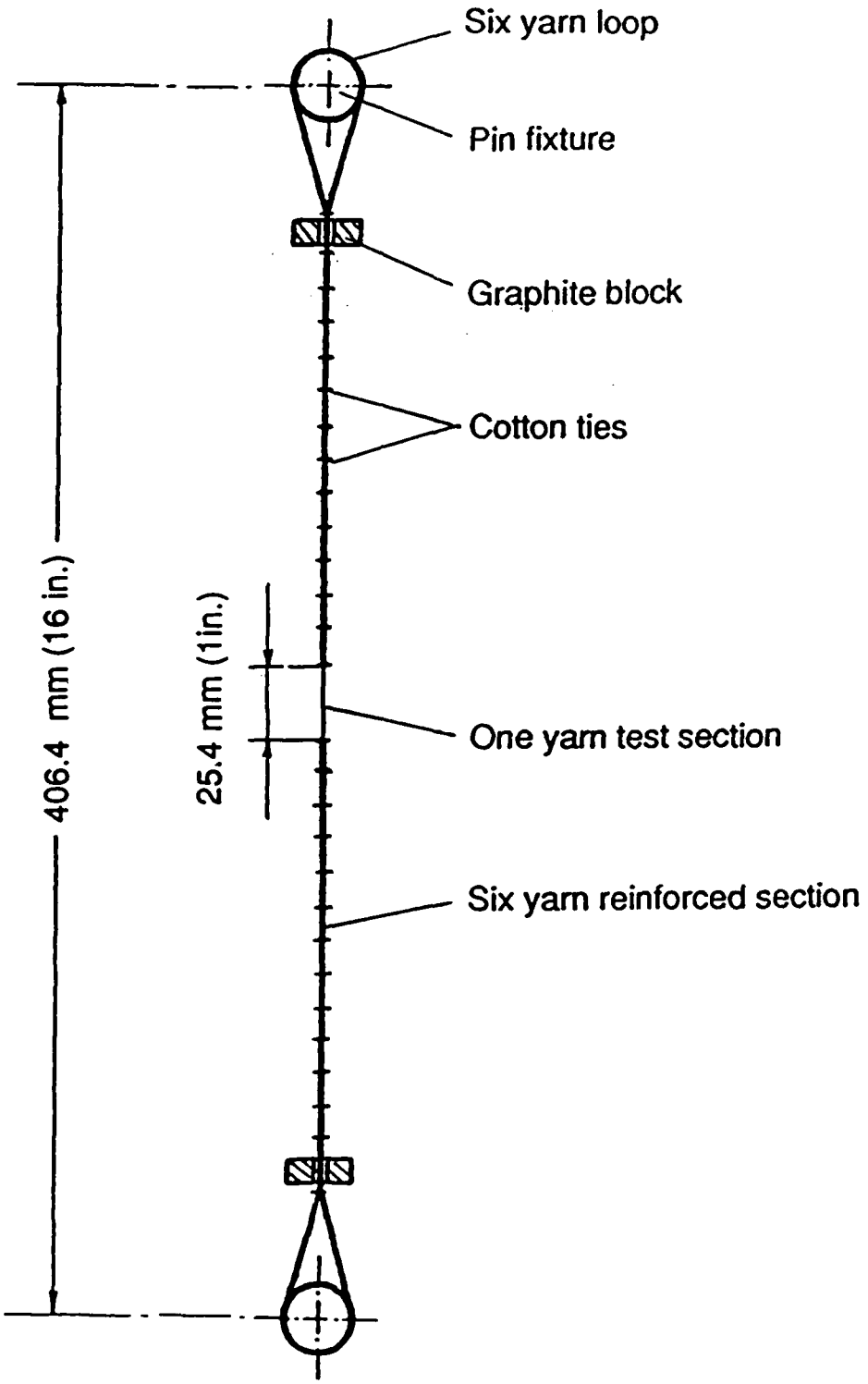


Fig. 1. Diagram of a uniaxial creep specimen. A single carbon yarn makes up the test section and extends the length of the specimen.

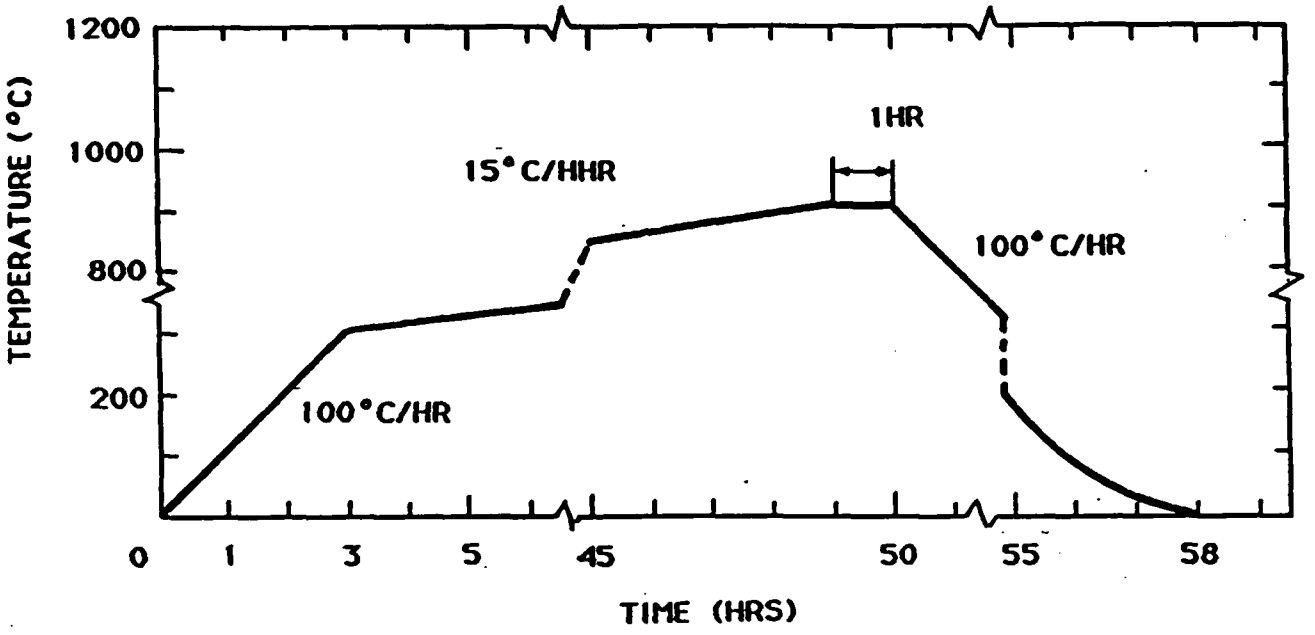
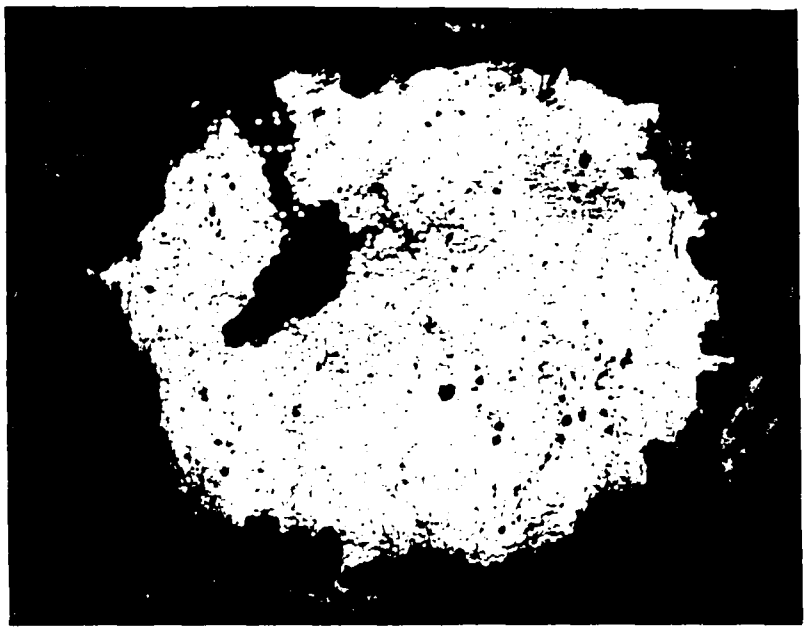


Fig. 2. Heating schedule for the carbonization of the creep specimen .



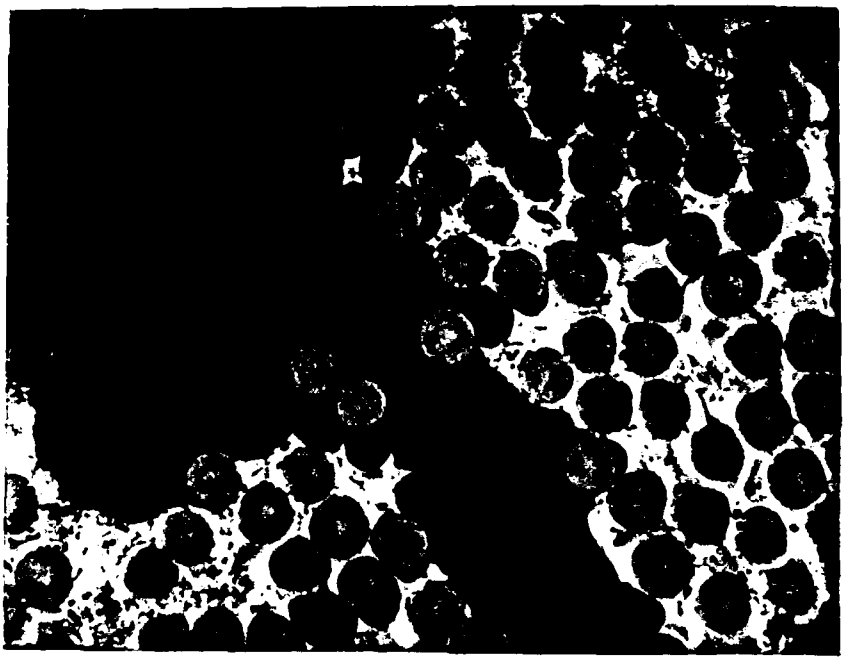
(a)

100um

Fig. 3. (a) Photograph (130x) of a cross-section of a carbonized test section. Note the unavoidable bubble pore in the upper left corner. (b) Magnified view (670x) showing the bonded filaments. (c) Magnified view (750x) showing the bonded filaments at the edge of the bubble pore.

(b)

20um



(c)

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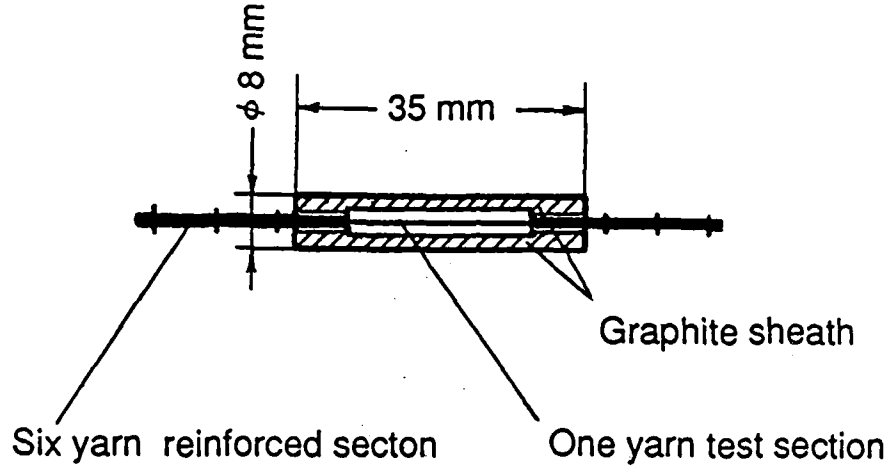


Fig. 4. Diagram of the graphite sheath used to cover the specimen's test section during the impregnation and carbonization of a 'dry' specimen.

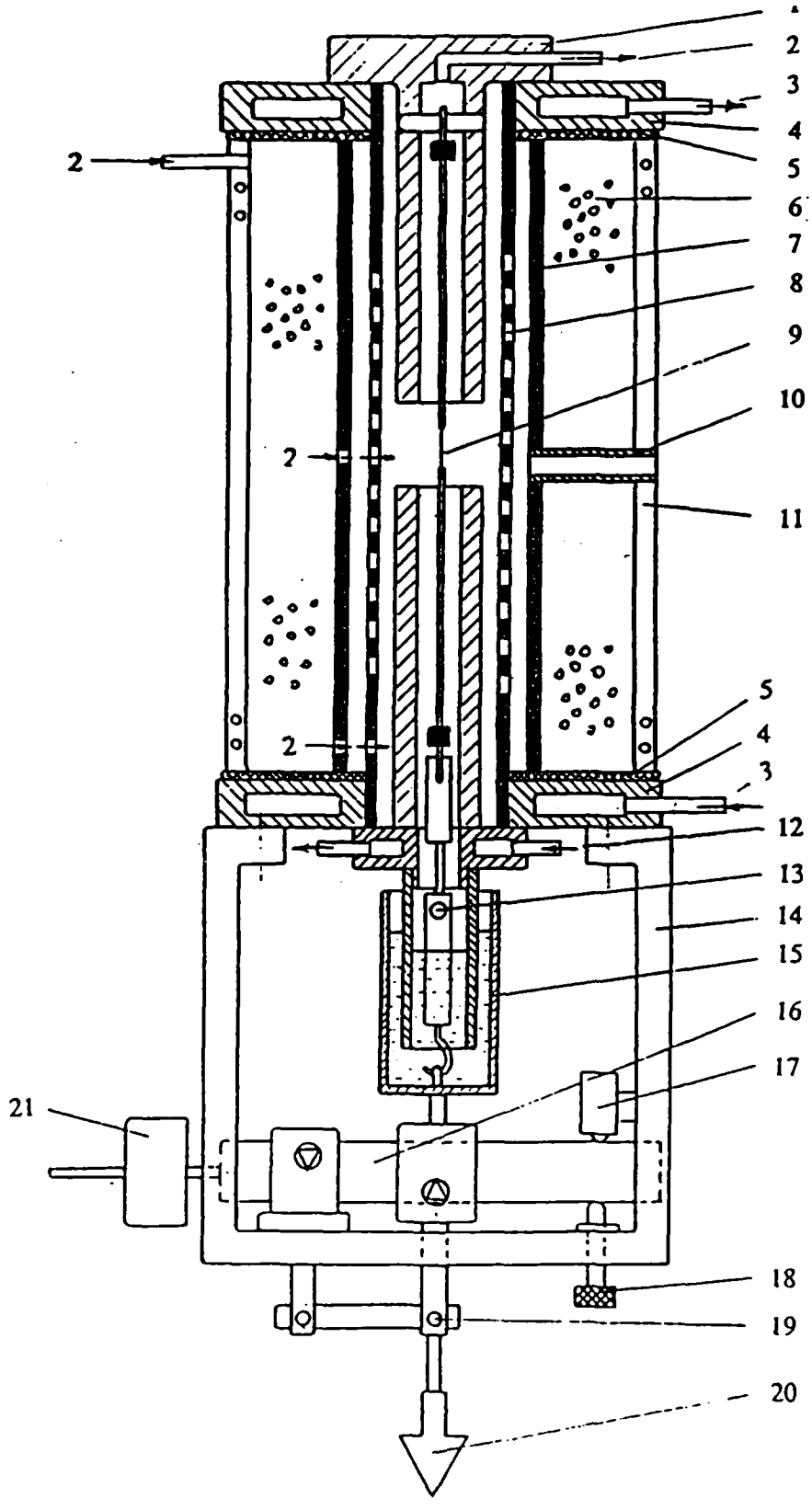


Fig. 5. Diagram of the high temperature furnace and the loading apparatus used for the creep experiments. (See the following page for number identification.)

1. Upper graphite test fixture (with pin to support test specimen)
2. Helium Inlets/outlet
3. Water Coolant Inlet/Outlet and Electrical Power Terminal
4. Upper/Lower Bulkhead Assembly
5. Electrical Insulator
6. Graphite Felt Pad Thermal Insulation
7. Graphite Shield
8. Graphite Heating Element
9. Uniaxial Creep Specimen
10. Window for Optical Pyrometer
11. Outer Furance Shell (water cooled by internal piping)
12. Lower Water Cooled Jacket
13. Specimen-Rotating Beam Linkage (including universal joint)
14. Loading Frame
15. Water Seal
16. Loading Beam
17. LVDT
18. Screw Support
19. Guide Link
20. Dead Weight Load
21. Counter Balance

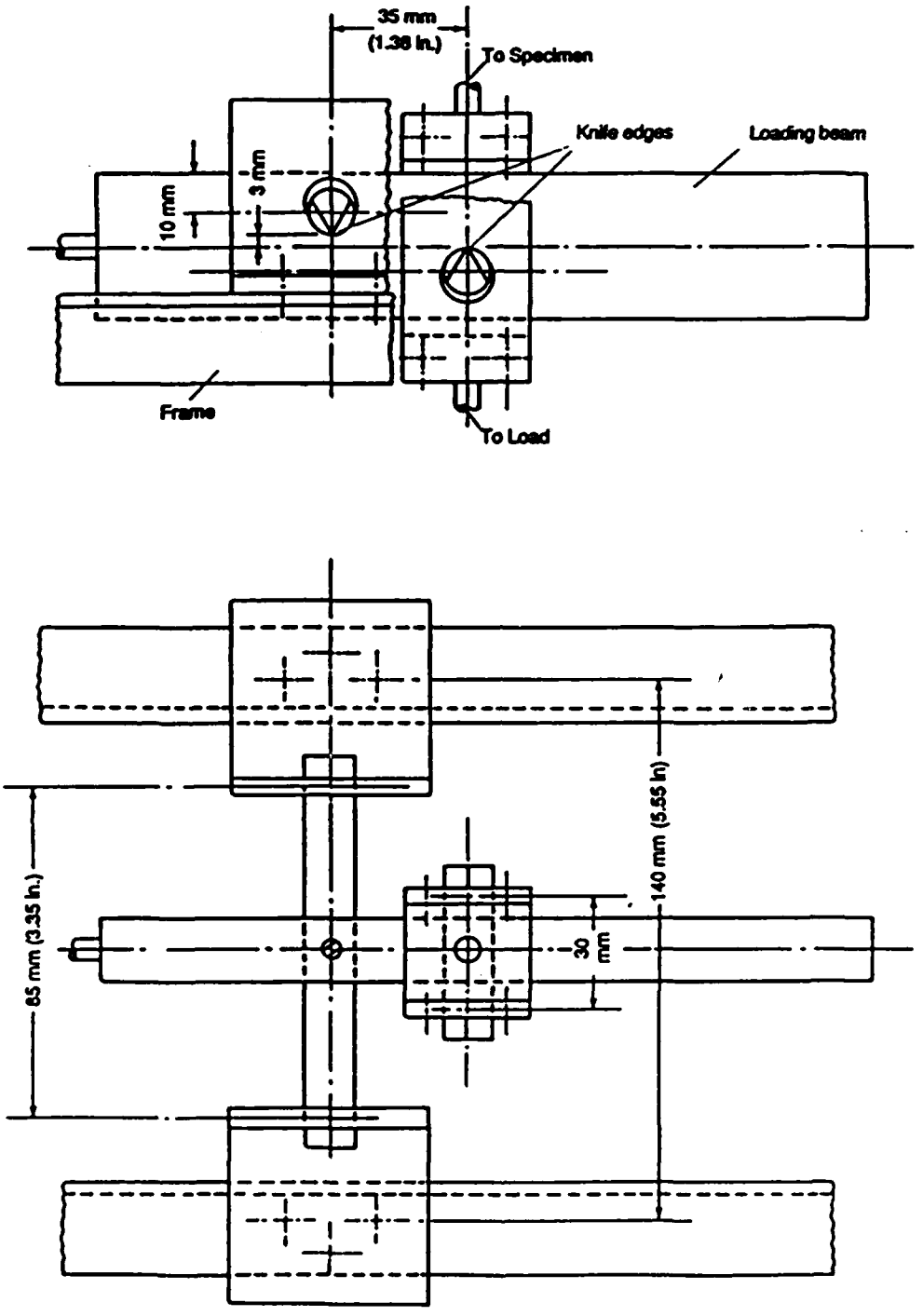


Fig. 6. Diagram showing the details of the loading beam. Knife edges were used to maintain alignment.

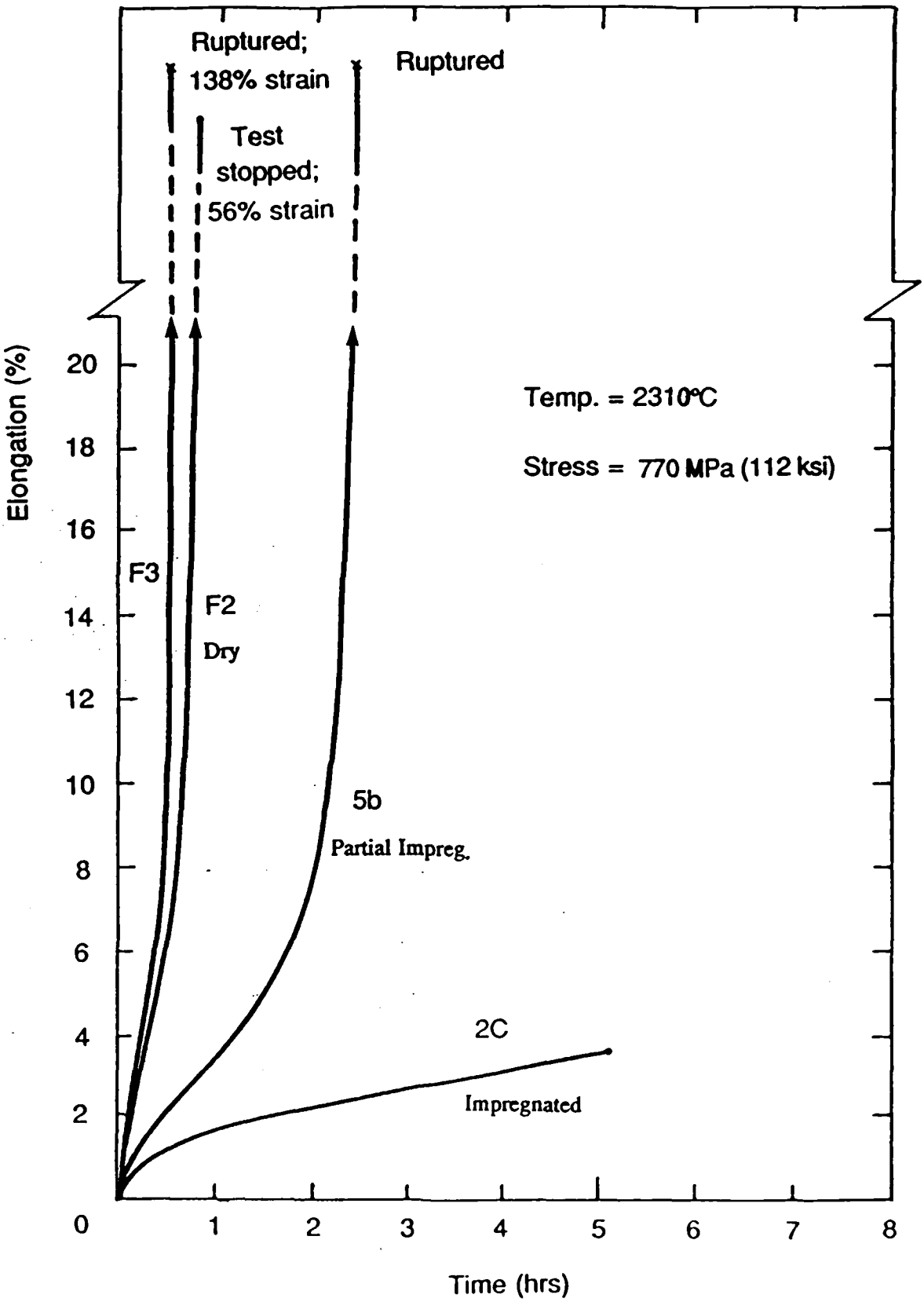
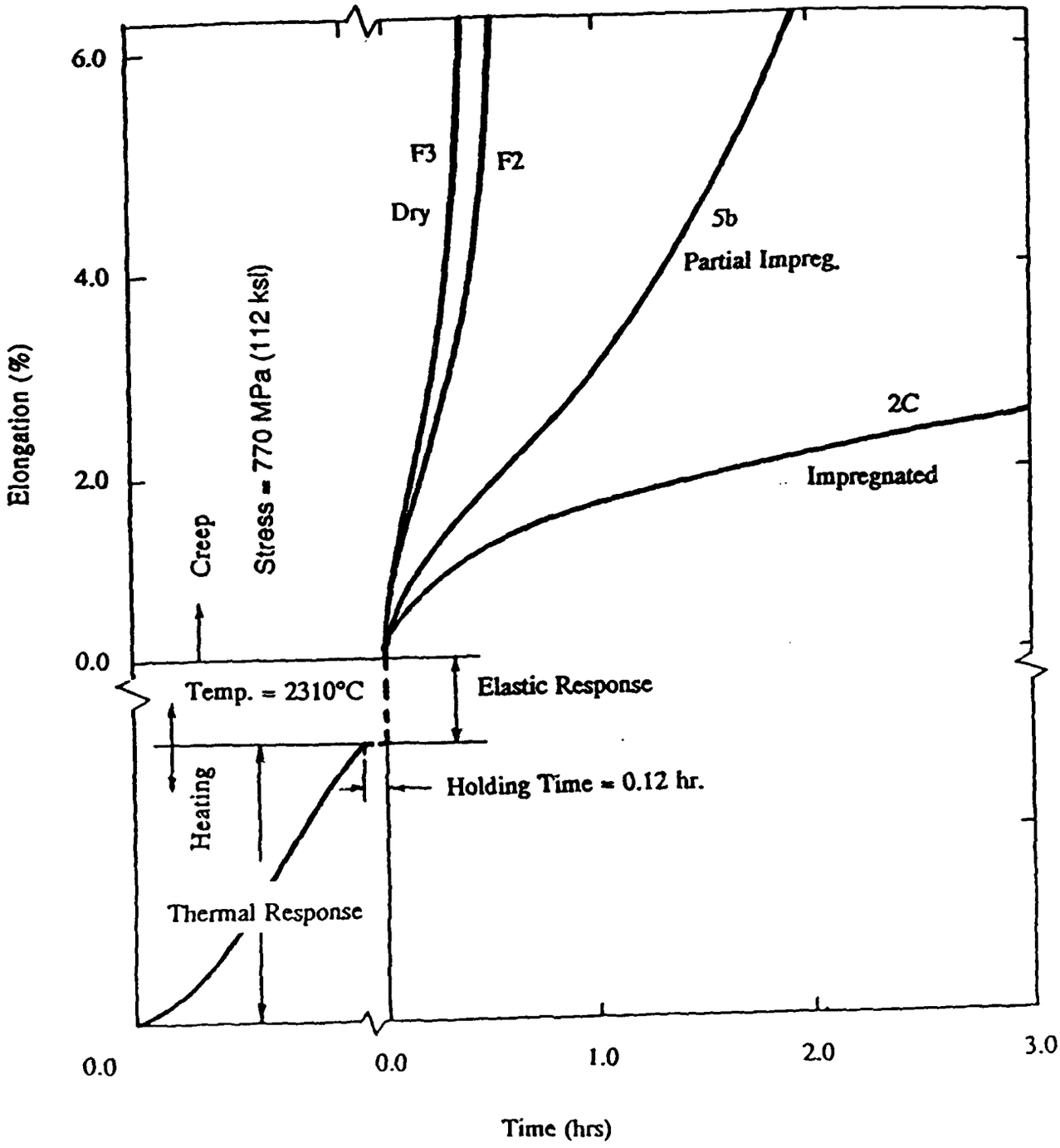


Fig. 7. (a) Elongation vs. Time plot showing the creep responses of the 'dry', partially impregnated, and fully impregnated specimens. (b) Enlarged view (see next page) of the initial responses as well as the pre-creep history of the specimens.


6.0
4.0
2.0
0.0
Elongation (%)
F3
F2
Dry
5b
Partial Impreg.
2C
Impregnated
Creep
Stress = 770 MPa (112 ksi)
Temp. = 2310°C
Elastic Response
Heating
Holding Time = 0.12 hr.
Thermal Response
0.0
0.0
1.0
2.0
3.0
Time (hrs)

(b)

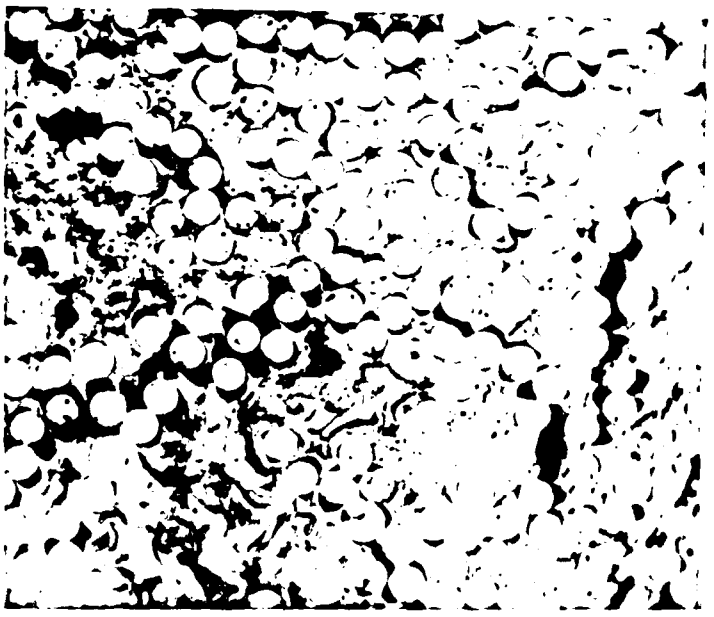


Fig. 8. Post-creep photograph of the test section of specimen 2C. There appears to be a higher percentage of debonded filaments than in the pre-creep photographs. (670x)

Table 1

Temperature = 2310°C Net Fiber Stress = 770 MPa (112 ksi)

Dry Bundle

Specimen F2: 56% strain when test was stopped after 0.73 hrs.

Specimen F3: 138% strain after 0.39 hrs and after rupture occurred.

Partial Impregnated

Specimen 5b: 18.8% strain after 2.35 hrs before rupture occurred.

Full Impregnated

Specimen 2C: 3.6% strain when test was stopped after 5.9 hrs.

Table 1. Summary of the data from fig. 7.

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